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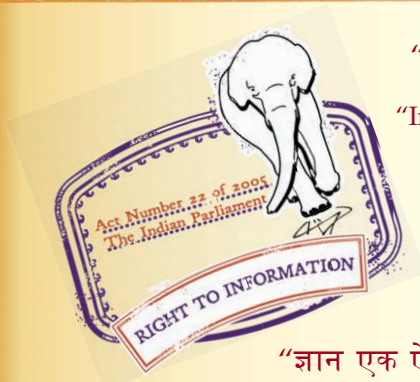
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# *Indian Standard*

## METHOD FOR EVALUATION OF STRENGTH AND SHADE OF NAPHTHOL

( First Reprint MARCH 1971 )

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# Indian Standard

## METHOD FOR EVALUATION OF STRENGTH AND SHADE OF NAPHTHOL

( First Reprint MARCH 1971 )

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# *Indian Standard*

## METHOD FOR EVALUATION OF STRENGTH AND SHADE OF NAPHTHOL

### 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 30 December 1968, after the draft finalized by the Dyestuffs Sectional Committee had been approved by the Textile Division Council.

**0.2** Naphthols are marketed in different strengths. The method prescribed in this standard for determining the strength of naphthols would be useful for assessing both the strength and the shade of the naphthol against mutually accepted standard. Unlike other types of dyes, the exhaustion of naphthols from the naphtholating bath is comparatively less. In order to reduce the chances of error in evaluating the strength of naphthols, two additional exhaust dyeings are recommended instead of a single dyeing and the average of the three dyeings is determined.

**0.2.1** Since it is equally important to evaluate the strength of naphthols by analytical methods, these methods have been included in IS : 4471-1967\*.

**0.3** This standard contains clause 3.1 which calls for agreement between the buyer and the seller and which permits the buyer to use his option for selection to his requirements.

**0.4** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

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### 1. SCOPE

**1.1** This standard prescribes a method for determination of strength of naphthols listed in Table I by dyeing test.

### 2. SAMPLING

**2.1** All the containers of the naphthol of same designation and strength delivered to a buyer against a despatch note shall constitute a **lot**.

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\*Methods for determination of strength of naphthols (azoic coupling components) (gravimetric and volumetric methods).

†Rules for rounding off numerical values (*revised*).

TABLE 1 GENERAL INFORMATION ABOUT DYEING OF NAPHTHOLS

(Clauses 1.1 and A-4.1.1)

SL. No.	COLOUR INDEX* DESIGNATION (COMMON COMMERCIAL NAME)	COLOUR INDEX No.	DEPTH OF SHADE	FOR NAPHTHOL DISSOLUTION, NAPHTHOL OF		FINAL CONCENTRATIONS IN NAPHTHOLATING BATH			COLOUR INDEX OF DIAZO COMPONENT (COMMON COMMERCIAL NAME)		CONCENTRATION OF BASE IN DEVELOPING BATH	pH OF DEVELOPING BATH	PERCENT NITRIC VALUE, ABOUT
				TRO	NaOH	TRO	NaOH	NaCl	Diazo Component	Molecular Weight			
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)
			g/l	ml/g	ml/g	ml/l	ml/l	ml/l			g/l		
i)	C. I. Azoic Coupling Component 2 (Naphthol AS)	C.I. 37505	2.0	3	1.5	8	6	20	C.I. Azoic Diazo Component 13 (Fast Scarlet RC)	204.5	1.5	4.5	80
ii)	C. I. Azoic Coupling Component 17 (Naphthol AS-BS)	C.I. 37515	1.0	3	1.5	8	6	20	C.I. Azoic Diazo Component 13 (Fast Scarlet R)	204.5	1.2	4.5	80
iii)	C. I. Azoic Coupling Component 4 (Naphthol AS-BO)	C.I. 37560	1.0	3	2	8	6	20	C.I. Azoic Diazo Component 5 (Fast Red B)	168	1.2	4.5	95

iv)	C. I. Azoic Coupling Component 18 (Naphthol AS-D)	C.I. 37520	2·0	3	5·1	8	6	20	C.I. Azoic Diazo Component 13 (Fast Scarlet R)	204·5	1·2	4-5	80
v)	C. I. Azoic Coupling Component 8 (Naphthol AS-TR)	C.I. 37525	1·0	3	2	8	6	20	C.I. Azoic Diazo Component 11 (Fast Red TR)	178	1·2	5-5	80
vi)	C. I. Azoic Coupling Component 5 (Naphthol AS-G)	C.I. 37610	2·0	3	2·5	8	6	15	C.I. Azoic Diazo Component 44 (Fast Yellow GC)	164	1·2	4-5	80
vii)	C. I. Azoic Coupling Component 13 (Naphthol AS-SG)	C.I. 37595	2·0	3	1·0	8	7	—	C.I. Azoic Diazo Component 5 (Fast Red B)	168	1·2	4-5	95
viii)	C. I. Azoic Coupling Component 15 (Naphthol AS-LB)	C.I. 37600	1·0	3	0·5	8	6	20	C.I. Azoic Diazo Component 2 (Fast Orange GC)	164	0·6	4-5	80

\*Colour Index (1956), Ed 2, Society of Dyers and Colourists, UK and American Association of Textile Chemists and Colourists, USA.

NOTE — Quantities mentioned under column (11) are based on the nitrite value of column (13).

**2.2** Unless otherwise agreed to between the buyer and the seller the number of containers to be selected at random from the lot shall be as given below:

<i>Lot Size</i>	<i>Sample Size</i>
Up to 25	2
26 „ 50	3
51 „ 150	5
151 and above	7

**2.3** From each container draw small quantities of dye by suitable sampling instrument from at least 3 different parts and mix them thoroughly to get a composite sample of desired weight.

### **3. STANDARD NAPHTHOL**

**3.1** The standard sample of naphthol against which the strength and shade of naphthol under test is evaluated, shall be as agreed to between the buyer and the seller.

### **4. QUALITY OF REAGENTS**

**4.1** Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

### **5. EVALUATION OF STRENGTH**

**5.1** Out of the sets of conditions of test laid down in Table 1, choose the set of conditions applicable to the naphthol under test.

**5.2** Prepare dyeing of the recommended depth of shade of naphthol under test by following the procedure given in Appendix A. Prepare simultaneously additional dyeing of the naphthol under test with the depth of shade 90 percent of the first.

**5.3** Simultaneously, prepare dyeings of different depths of shade of standard naphthol by following the procedure in Appendix A.

NOTE — The depths of shade of dyeings of standard naphthols should be so arranged that the difference between two consecutive dyeings is the same and the dyeings are well distributed on either side of the recommended depth of shade ( see Table 1 ), taking into consideration the expected strength of the sample.

**5.4** Carry out second set of dyeings with fresh hanks in the exhaust liquor of the above dyeings by following the procedure given in **A-4.5**.

**5.4.1** Carry out third set of dyeings with fresh hanks in the exhaust liquor obtained as in **5.4** by following the procedure given in **A-4.5.1**.

**5.5** Compare the dyeings obtained as in **5.2** and the dyeings obtained as in **5.3** ( *see* Note ). Select dyeing of the standard naphthol which exactly matches with one of the dyeings of the naphthol under test. Note the depths of the shade of the dyeings which match exactly.

NOTE — The dyeing should be spread out properly and compared in the north light.

**5.6** Calculate the strength of naphthol under test by comparing the first set of dyeings by the following formula:

$$S = \frac{A}{B} \times 100$$

where

$S$  = strength of naphthol in percent,

$A$  = depth of shade in grams per litre of the dyeing of standard naphthol, and

$B$  = depth of shade in grams per litre of the dyeing of naphthol under test comparing with that of standard.

**5.6.1** Similarly calculate the strength of naphthol under test from the other two sets of exhaust dyeings.

**5.7** Calculate the average of the three values obtained as in **5.6** and **5.6.1**.

## 6. REPORT

**6.1** Report the value obtained as in **5.7** as the strength in percent of the naphthol under test as compared to the standard.

**6.1.1** Report also the shade of the naphthol under test in comparison with the shade of the standard.

NOTE — The remarks regarding shade would hold good only for the specific combination of naphthol and base mentioned in Table 1.

## APPENDIX A

( *Clauses 5.2, 5.3, 5.4 and 5.4.1* )

### DYEING PROCEDURE

#### A-1. APPARATUS

**A-1.1 Dye Vessels** — Porcelain, glass or stainless steel beakers or dry vessels of 250 to 300 ml capacity.

NOTE — For diazotization, only porcelain or glass beakers should be used.

## A-1.2 Watch-Glass

## A-1.3 Pipettes Graduated

# A-2. DYEING ASSISTANTS

**A-2.1 Water** — Distilled water ( *see* IS : 1070-1960\* ) shall be used in the preparation of dye-baths.

NOTE — For rinsing, water having hardness of not more than 50 ppm expressed as calcium carbonate may be used.

**A-2.2 Sodium Hydroxide Solution** — 430 g/l or 70° Tw.

**A-2.3 Turkey Red Oil** — 50 percent solution ( *v/v* ).

## A-2.4 Soap-Neutral

**A-2.5 Soda Ash** — ( *see* IS : 251-1962† ).

**A-2.6 Sodium Chloride Solution** — 10 percent ( *w/v* ).

**A-2.7 Hydrochloric Acid** — 32° Tw.

**A-2.8 Sodium Nitrite** — Solid ( 98 percent ).

**A-2.9 Sodium Acetate** — Solid ( 98 percent ) ( *see* IS : 557-1954‡ ).

**A-2.10 Acetic Acid** — 50 percent ( *w/v* ).

# A-3. PREPARATION OF HANKS

**A-3.1 Test Hanks** — Hanks shall be of scoured, bleached, unmercerized cotton yarn having no finishing chemical or blueing agent. Each hank shall weigh  $10 \pm 0.1$  g.

NOTE — Any yarn normally used in the laboratories for carrying out trials or yarn of the following requirements is suitable for this test:

Count	10 tex $\times$ 2 ( or 60 <sup>s</sup> /2 )
Twist	750/m
Cuprammonium Fluidity	Not more than 5 rhes

**A-3.2 Preparation of Test Hanks** — The hanks shall be treated in boiling water for 10 minutes and squeezed evenly to contain approximately its own weight of water, cooled and entered in the naphtholating bath.

# A-4. PROCEDURE

## A-4.1 Preparation of Naphthol Solution

**A-4.1.1** Weigh accurately 5.0 g of naphthol under test. Paste it thoroughly with Turkey red oil. Add requisite quantity of sodium

\*Specification for water, distilled quality ( *revised* ).

†Specification for soda ash, technical ( *revised* ).

‡Specification for sodium acetate, technical and photographic. ( Since revised ).

hydroxide solution ( *see* Table 1 ). Add 100 ml of water near boil and boil the whole solution till it becomes clear. Dilute the solution to 500 ml with water containing 1 ml/l each of sodium hydroxide and Turkey red oil solutions.

NOTE 1 — C. I. Azoic Coupling Component 13 ( Naphthol AS-SG ) is dissolved by following the procedure given below:

‘ Paste 5.0 g of naphthol with equal amount of Turkey red oil. Add to it 5 ml of hot water and 5 ml of sodium hydroxide solution. Heat the paste for 15 minutes and then add 30 ml of boiling water. Boil the solution and add 40 ml of boiling water. Boil the solution again. Dilute the clear solution to 500 ml with water containing 1 ml/l each of Turkey red oil and sodium hydroxide solution. ’

NOTE 2 — In case of C. I. Azoic Coupling Component 8 ( Naphthol AS-TR ) 200 ml of water is used for naphthol dissolution. Also the caustic soda is added to the boiling suspension of naphthol in water.

**A-4.1.2** Similarly prepare solution of the standard naphthol by following the procedure given in **A-4.1.1** but taking standard naphthol instead of naphthol under test.

**A-4.2** For naphtholation, developing, and soaping, the material to liquor ratio shall be 1 : 20.

#### **A-4.3 Naphtholation ( for $10 \pm 0.1$ g Hank )**

**A-4.3.1** Take in each dye-vessel required quantity of water ( *see* Note ). Add the necessary amount of Turkey red oil and sodium hydroxide ( subtracting the amount of sodium hydroxide and Turkey red oil coming with naphthol solution ). Add the necessary amount of naphthol solution under test in one dye-vessel so that the final concentration of the naphthol under test shall be as given in Table 1. Add the necessary amount of sodium chloride solution with stirring. Similarly add in other dye-vessel solution of naphthol under test so as to give 90 percent depth of shade of first dyeing. Enter the wetted and squeezed hanks into the dye-vessels and naphtholate for 30 minutes at room temperature. Turn the hanks so as to obtain level naphtholation. After naphtholation take out the hanks and squeeze them evenly. Preserve the naphtholating bath for carrying out exhaust dyeing ( *see* **A-4.5** ).

NOTE — The volume of water should be calculated taking into consideration the volume of sodium hydroxide, Turkey red oil, sodium chloride and naphthol solution taken for naphtholation.

**A-4.3.2** Similarly, prepare naphtholated hanks with the solution of standards naphthol in different depths ( *see also* Note under **5.3** ).

NOTE — The naphtholation with the solutions of standard naphthol and the naphthol under test should be done simultaneously.

**A-4.4 Developing** — Prepare a developing bath in water containing suitable diazotized fast base ( *see* Table 1 and Notes below ). Develop all the naphtholated hanks obtained as in **A-4.3.1** and **A-4.3.2** in the same

developing bath at room temperature for 30 minutes with frequent stirring. Wash the dyed hanks thoroughly with water.

NOTE 1 — The details of the diazotization are given in Appendix B.

NOTE 2 — The pH of the developing bath can be adjusted to the desired value by addition of acetic acid or sodium bicarbonate solution as the case may be.

NOTE 3 — In case of C. I. Diazo Component 11 ( Fast Red TR Base ) sodium acetate crystals 10 g/l are added to the developing bath before use.

NOTE 4 — For developing bath of C. I. Azoic Coupling Component 5 ( Naphthols AS-G ) an addition of 4 ml/l of acetic acid ( 50 percent ) is made over and above the normal quantity of alkali binding agent.

**A-4.5** Make up the volumes of each naphtholating bath obtained in **A-4.3.1** and **A-4.3.2** to 200 ml with water containing 1 ml/l each of sodium hydroxide solution and Turkey red oil. Carry out the naphtholation of the fresh hanks and develop the naphtholated hanks in the fresh baths.

**A-4.5.1** Repeat naphtholation and developing of fresh hanks as per method given in **A-4.5**.

**A-4.6 Soaping** — Soap all the three sets of dyeings ( see **A-4.4**, **A-4.5** and **A-4.5.1** ) together at boil for 30 minutes in a bath containing 3 g/l of soap and 2 g/l of soda ash. Wash thoroughly and dry.

## **APPENDIX B**

*( Note under clause A-4.4 )*

### **DIAZOTIZATION RECIPES FOR FAST BASES**

#### **B-1. C.I. AZOIC DIAZO COMPONENT 13 ( FAST SCARLET RC BASE )**

**B-1.1** Paste 10 g fast scarlet-RC base with a mixture of 10 ml hydrochloric acid and 10 ml cold water. Add to this 200 ml cold water and stir to dissolve. Cool the solution to 10 to 15°C. Add to this quickly whilst stirring 4 g sodium nitrite dissolved in 25 ml cold water. After 20 to 30 minutes neutralize the solution with 7.5 g sodium acetate dissolved in 25 ml cold water. Add finally the alkali binding agent 5 ml acetic acid ( 50 percent ).

Diazotizing Temperature: 10 to 15°C.

**B-2. C.I. AZOIC DIAZO COMPONENT 5 (FAST RED B BASE)**

**B-2.1** Paste 10 g fast red-B base with 15 ml hot water and 5 g sodium nitrite. After the nitrite is completely dissolved, cool the paste to 15°C and add in small quantities with vigorous stirring to a mixture of 200 ml cold water and 17.5 ml hydrochloric acid. Allow to stand for 30 minutes with frequent stirring. Filter the solution and neutralize with 8.5 g sodium acetate dissolved in about 25 ml cold water. Finally add the alkali binding agent 7.5 ml acetic acid (50 percent).

Diazotizing Temperature: 15°C.

**B-3. C.I. AZOIC DIAZO COMPONENT 11 (FAST RED TR BASE)**

**B-3.1** Dissolve 10 g fast red-TR base in a mixture of 200 ml cold water and 10 ml hydrochloric acid. Cool the solution to 10°C whilst vigorously stirring and fairly slowly 4 g sodium nitrite dissolved in 25 ml cold water. After 20 to 30 minutes neutralize with 7.5 g sodium acetate dissolved in about 25 ml cold water. Add the alkali binding agent 1.5 ml acetic acid (50 percent). Add further 10 g sodium acetate to the dye-bath before use.

Diazotizing Temperature: 10°C.

**B-4. C.I. AZOIC DIAZO COMPONENT 44 (FAST YELLOW GC BASE)**

**B-4.1** Paste 10 g fast yellow-GC base with 50 ml hot water and 12 ml hydrochloric acid. Add 150 ml cold water and cool to 10°C. To the solution add 5 g sodium nitrite dissolved in about 25 ml cold water with stirring. After 15 to 20 minutes neutralize with about 10 g sodium acetate dissolved in 25 ml cold water. Then finally add the alkali binding agent 7.5 ml acetic acid (50 percent).

Diazotizing Temperature: 5 to 10°C.

**B-5. C.I. AZOIC DIAZO COMPONENT 2 (FAST ORANGE GC BASE)**

**B-5.1** Paste 10 g fast orange-GC base with 50 ml hot water and 12 ml hydrochloric acid. Add 150 ml cold water to dissolve the paste. Cool to 10°C. Add with stirring 5 g sodium nitrite dissolved in about 25 ml cold water. After 15 to 20 minutes neutralize with 10 g sodium acetate dissolved in about 25 ml cold water. Finally add the alkali binding agent 7.5 ml acetic acid (50 percent).

Diazotizing Temperature: 10°C.

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